to perform time-consuming calculations in a fraction of the time required on a single machine. In most cases, users can now take home their processed data within hours of the completion of their experiment rather than take it home and struggle for days to process it themselves. In the future, user interface improvements to this system will open up this capability to all users with a need for processing power.

Improvements and new features have also been added to the CCD acquisition program used by the majority of SRI-CAT users for acquiring CCD data. It is now possible for users to control cameras from five different manufacturers through the same user interface—all seamlessly integrated into the standard EPICS control system. Additional data acquisition modes have been added to make the system more useable to a greater number of people.

2.3 X-ray Optic Fabrication and Metrology

The CAT members at the APS continued to make requests for x-ray optics from the staff of the Optics Fabrication and Metrology (OFM) Group. As detailed below, these requests have come in four areas: metrology, thin film deposition, crystal fabrication, x-ray characterization, and mirror design and consulting. Recent highlights include:

- Differential deposition of gold for K-B mirrors was perfected to achieve 0.90 microrad rms deviation over 60 mm from an ideal elliptical surface starting from a spherically shaped silicon substrate.
- Fabrication of a Si crystal designed for sagittal focusing in a bender with a thickness of 0.9 mm over a diffraction area of 53 mm by 76 mm and with a thickness variation of only 5 microns over the entire diffraction area was achieved.
- Parabolic refractory lenses made by extrusion and having a 0.2 mm wall thickness were fabricated very inexpensively and tested at 81 keV to show effective focusing. Such lenses were assembled into a variable focus unit. Subsequently lenses were obtained for which the wall thickness was reduced to 0.1 mm.
- Polishing of silicon to a roughness of 0.09 nm (rms) was accomplished in-house with chemical mechanical polishing.
- Stitching interferometry, which promises to provide precise figure error data for large mirrors over an area, was demonstrated
- A double-multilayer monochromator was made and put into service on beamline 2-BM (Chu et al., 2002).
- Etching of diamond monochromators to remove strain was found to be efficacious (Maj et al., 2002).

2.3.1 X-ray Optics Metrology Laboratory

Measurement Requests

During the last fiscal year, we handled a total of 110 metrology measurement requests out of which 56 originated from the CATs. These included beamline mirrors, multilayers optics, and mirror bender systems. Metrology requests were filled for the following CATs: BESSRC, Bio, CMC, DND, GSECARS, MHATT, SER, SBC, SGX, SRI, and UNI. Requests from non-APS users originated from SNS/IPNS, Fermi Laboratory, and others. In addition, the laboratory continues to provide measurement data to the OFM Group's polishing facility, thus indirectly supporting the users activities. Such measurements are also used to monitor and improve our polishing capability, which equally benefits the APS users.

Instrumentation Upgrades and Developments

With the advances in synchrotron radiation sources and research, tolerances on x-ray mirror quality are becoming tighter. There is now demand for mirrors with surface roughness and slope error on the order of ≤1 Å rms, and ≤1 μrad rms, respectively. Metrology of mirrors with such a tolerance level is expected to be challenging, particularly with respect to the APS long trace profiler (LTP II), which has a sensitivity that is limited to 0.5 μrad. Moreover, one of our major ongoing efforts relates to the development of an elliptically shaped K-B microfocusing mirror using a differential deposition technique (Ice et al., 2000). The LTP II data continue to be essential in the optimization of the shape and thickness of the differentially deposited films on K-B mirror substrates. Progress towards achieving diffraction-limited focusing will require even highly optimized differentially deposited thin film. This, along with efforts in developing coherence preserving optics, will likely push the measurement accuracy far below the limits of our current LTP. Therefore, a number of steps are being considered to enhance the instrument performance. One of the most crucial optical components is the Fourier transform (FT) lens. The existing FT lens suffers from aberration impacting the accuracy of measurements, particularly those concerning strong aspheric surfaces. We are pursuing obtaining an improved lens for the LTP. Since K-B mirrors are generally much shorter than regular x-ray mirrors, a compact LTP is being constructed. The ultimate resolution is expected to be better for the smaller unit.

Development of a Stitching Interferometry System for Large X-ray Mirrors

To enhance the metrology measurements and complement the LTP II instrument, a stitching interferometry system for evaluating large x-ray mirrors is being developed in collaboration with Michael Bray at MB-Optique Inc, (Assoufid et al., 2002a). The system has the potential of providing a full 3-D surface profile with nanometer resolution, and the data can be used, for example, to provide feed back for surface error correction during mirror polishing. The data can also be used for simulation purposes.

The system is being developed around an existing laboratory laser interferometer (a WYKO-6000 surface profiler), a 1-m-long high-accuracy translation stage/rail, a tilt platform, a mirror mount system, and the stitching computer code provided by M. Bray.

The measurement procedure consists of acquiring N overlapped subaperture measurements until the whole mirror surface is covered. At the end of the measurement sequence, the full 3-D surface is constructed by stitching together the submeasurements, using the stitching computer code.

Preliminary tests were performed on an actual silicon beamline mirror. The measurement required eight subaperture measurements with 41% overlap between adjacent subapertures, and each subaperture represented an average of ten phase measurements. Table 2.2 compares the resulting surface slope error value with that obtained with the LTP II. As one can see, the rms slope error values are in close agreement, but there is a large discrepancy in the peak-to-valley values, which is due to

Table 2.2. Comparison between LTP II and stitching interferometry data obtained on a silicon mirror over a 750-mm-long scan length. (Note that the LTP II data are the result of a one-dimensional trace along the mirror axis, while the stitching data are the result of measurement of a surface 750 mm long by 40 mm wide.)

Instrument/Measurement Technique	**rms Slope Error (μrad)**	**Peak-to-Valley (μrad)**
LTP II	2.0	11.2
Stitching Interferometry	1.6	70.3

a bad pixel in the stitched profile, thus giving an exaggerated peak.

With this work, we demonstrated that the technique can be easily adapted for evaluation of large synchrotron radiation mirrors, and the preliminary results were encouraging. However, there are still a few sources of errors that must be tackled in order to obtain reliable, large field-of-view images with the desired accuracy. These sources of errors pertain mainly to temperature control and system calibration. We are currently actively working on implementing the necessary improvements.

Applications of the Atomic Force Microscope

The atomic force microscope (AFM) has proven to be a very useful metrology tool and is now routinely used. The PSD data derived from the AFM topography measurements are very useful for comparison with those obtained from x-ray scattering measurements. A variety of samples were characterized for the users during the last fiscal year. These include thin films (Satyam et al., 2001), patterned deposition of metallic nanoclusters (Divan et al., 2001), zone plates, and superpolished silicon substrates.

Investigation of Polished Silicon Substrates Using X-rays

In addition to metrology at optical wavelengths, we began using x-rays as a means of characterizing polished substrates destined for x-ray use. The focus was essentially on silicon surfaces prepared in the OFM Group polishing facility. The intent of this work was to quantitatively assess the nature of the power spectrum of the surface as measured by x-rays and that would influence the x-ray scattering properties of optics made using such surfaces. To this end, x-ray scattering measurements were conducted at the C station of 1-BM beamline on three polished silicon samples with nominal roughness of 0.9, 1.6, and 10 Å rms, respectively, as measured using our WYKO TOPO-2D. Figure 2.21 shows the AFM surface topography images for the three silicon substrates. This study showed that the power spectra derived (see Fig. 2.22) from x-ray diffuse scattering data agree reasonably well with those obtained from the AFM surface topography measurements. The data also showed features with a size comparable to that of the colloidal particles used for polishing (65 Å) (Assoufid et al., 2002b).

a)

b)

c)

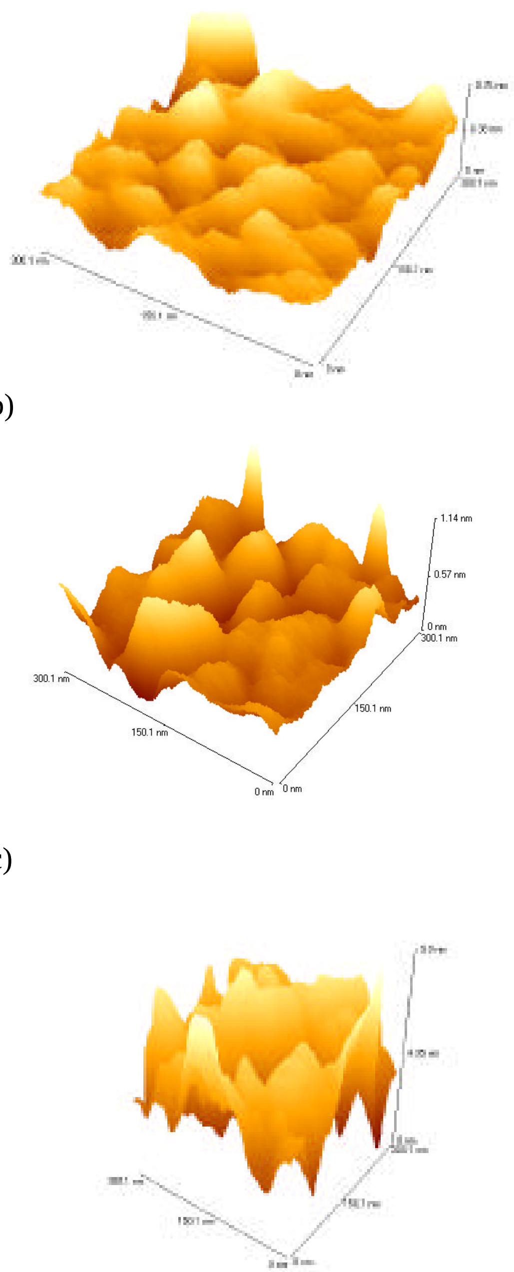

Fig. 2.21. Atomic force microscopy surface topography of three silicon substrates polished at the OFM Group facility. The three substrates have a surface roughness of a) 0.9, b) 1.6, and c) 10 Å rms, respectively, as measured with the WYKO TOPO-2D.

2.3.2 Deposition Laboratory

We have filled deposition requests from users including over 200 regular depositions and more than 450 mirrors and experimental samples since September 2000. Kirkpartrick-Baez grazing-incidence mirrors and multilayer x-ray optics are routinely fabricated at the deposition lab. Coatings for SRI-CAT x-ray lithography experiments are made in large quantities.

Graded Multilayers

Continuous efforts in laterally graded multilayers have been made after the initial success in tunable x-ray double-monochromator applications (Liu et al., 2001). Graded multilayers can now be grown in both the large and the small deposition systems. Using the large deposition system, the grading length can be expanded to 6 inches. Recently we have used the large deposition system to make graded multilayers for x-ray fluorescence detection applications. Figure 3.23 shows the measured d spacing and reflectivity as a function of lateral distance along a W/C graded multilayer made for Bio-CAT. The measurements were done at 6.5 keV on the Bio-CAT undulator x-ray beamline (via K. Zhang of Bio-CAT). The multilayers consists of 60 bilayers of a uniform W layer and a wedge-shaped C layer grown on a 100 mm × 100 mm × 2 mm Si substrate with a 0.7 nm rms roughness.

Selective Profile Coating/ Differential Deposition

Our experience in graded multilayers has been expanded into coating films with other selective thickness profiles. One application of this technique is to convert a cylindrical mirror to an elliptical one by differentially depositing Au films. In this application, a desired surface profile after Au coating on a cylindrical mirror should be the ideal surface figure of a focus ellipse. In the selective profile coating, the sputter source power is kept constant while the substrate is passed over a contoured mask at a constant speed. The mask is placed very close to the substrate level (within ~1.5 mm) on a shield can over the sputter gun (see Fig. 2.24). To determine the shape of the contour, we first measured the Au thickness distribution above the sputter gun at the substrate level. Figure 2.25 shows such a distribution of a Au film on a 4-inch-diameter Si wafer measured by using an ellipsometer. The growth was done without any masks nor substrate movements. The units in Fig. 2.25 are angstroms for the vertical axis and cm for the horizontal axes. The center directly above the target shows the largest thickness, as expected. A model has been developed to fit the measured thickness distribution for this stationary growth. The relative thickness weightings are then digitized at every point 1 mm apart for an area of 76 × 152 mm^2 directly above the sputter gun at the substrate level. When the substrate is moving across the shield can, the film

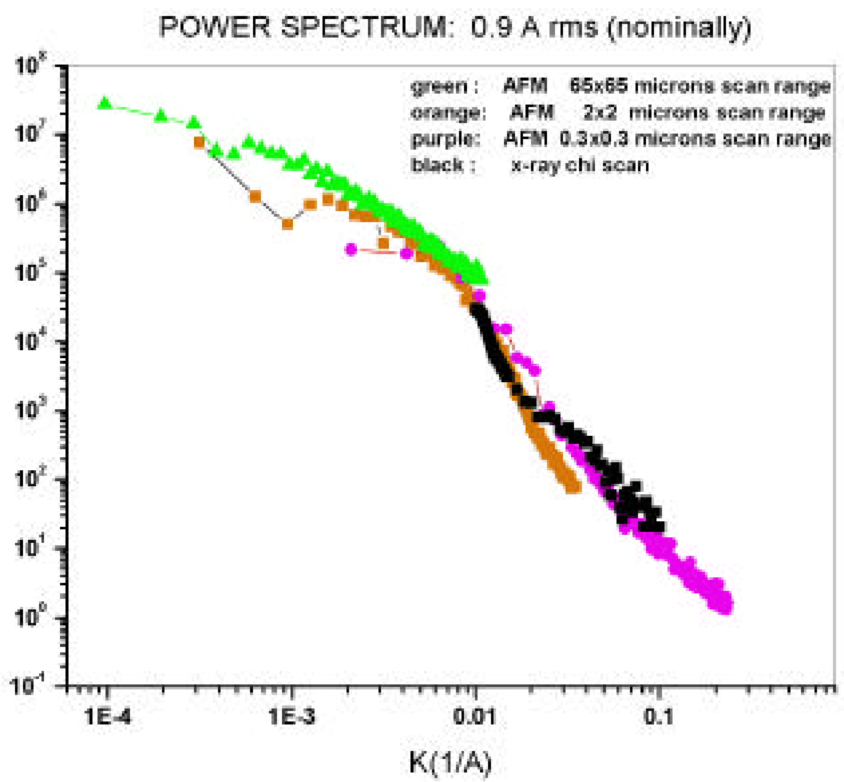

Fig. 2.22. Power spectral density (PSD) curve obtained from x-rays and AFM for 0.9 Å superpolished silicon.

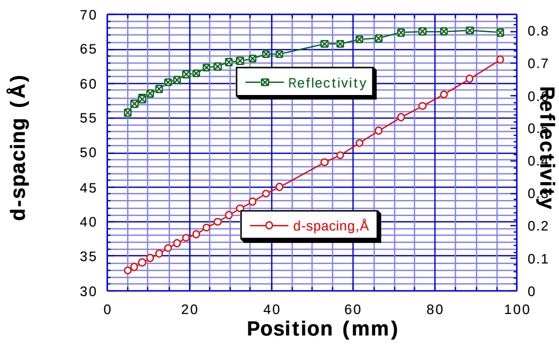

Fig. 2.23. Measured d spacing and reflectivity as a function of position along a W/C graded multilayer made for Bio-CAT. The measure-ments were done at 6.5 keV on the Bio-CAT undulator x-ray beamline (via K. Zhang of Bio-CAT).

thickness is directly proportional to the length of the opening on the can. By equaling the summation of relative weighting to the required relative thickness, a contour can be obtained for a desired thickness profile. A desired profile for changing a 90-mm-long cylindrical mirror into an elliptical one is shown as the "initial" required thickness in Fig. 2.26. With the contoured mask in place, a film with the desired thickness profile can be grown by passing the substrate over the mask during the deposition. By adjusting the speed and the number of passes of the substrate, a desired total film thickness can be achieved. The results to date have been encouraging.

After only one deposition, the surface figure of the mirror was already close to that of an ideal ellipse. As judged by LTP measure-ments, the difference from the ideal ellipse is shown in Fig. 2.26 as the required thick-ness "after one deposition." For over a 30 mm length, the mirror had a figure error of 0.47 microrad from an ellipse. This is beyond the known state-of-the-art which is 0.69 microrad over 20 mm. Very recently we have been able to extend this result to

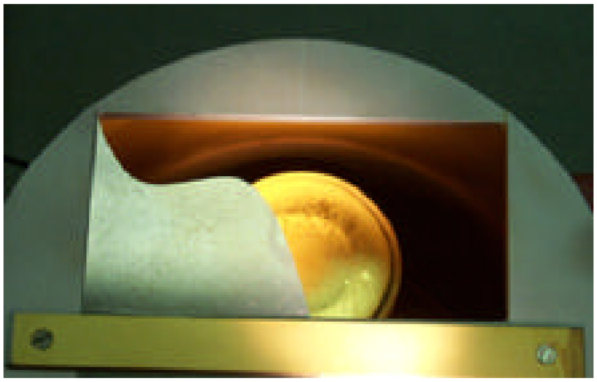

Fig. 2.24. A mask placed on top of the shield can above the Au target to achieve a desired Au thickness profile on a cylindrical mirror for ellipse focusing. The opening on the can top is 3" × 6". The 90-mm-long mirror was placed on a carrier with one end aligned to the far-left side of the mask and the other end towards the center. During the deposition, the mirror was moving across the mask so that the end which moves across the center receives the thickest deposition.

longer lengths and have acheived 0.90 microrad rms over 60 mm.

System Improvements

We have improved the large deposition system and maintained the facility in good working order. The shielding cans for all four sputter guns have been redesigned so that 6-inch-wide substrates can be coated in the large deposition system. The previous limit was 4 inches. We improved the transport system in the large deposition system by adding a cable-separator so that the previously crossed pulling cables no longer interfere with each other.

The ellipsometer has been upgraded so that automated measurements can be performed on 4" × 4" samples to obtain a film thickness profile. A Specs ion sputter-cleaning gun has been added to the large deposition system. It is useful for modifying the surface structure of a sample.

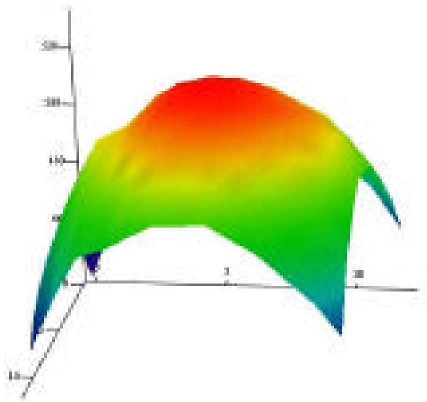

Fig. 2.25. Thickness distribution obtained from ellipsometer measurements of a gold film on a Si wafer placed directly above the Au target. The units are angstroms for the vertical axis and cm for the horizontal axes.

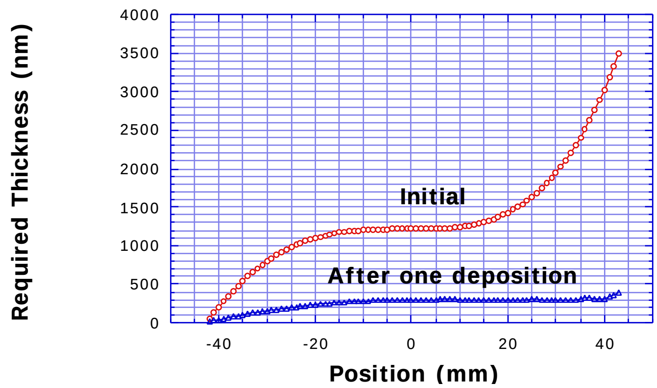

Fig. 2.26. Required thickness profile to achieve an ideal ellipse surface profile according to LTP measurements.

2.3.3 Fabrication Laboratory

The optics fabrication laboratories are the leading suppliers of x-ray difractive optical elements for APS beamlines. For the period of June 2000 – April 2002, 510 optical elements made of Si, Ge, Sapphire, MgO and different type of glasses have been fabricated. Elements were fabricated for other research centers as well, such as UOC, SNS, NWU, NIST. For example, a cryocrystal was made for Bio-CAT and for SBC. Both (111) and (100) versions have been fabricated and delivered.

Purchasing, installation, and learning how to properly operate the K&S Dicing saw was notable. It enhanced our capabilities for more precise cutting and dicing slabs and microstructures up to 5 mm thick and 200 mm in diameter. By using the unique technical capabilities of this cutter, we have fully fabricated six silicon and germanium substrates for x-ray analyzers. The machine is being widely used for fabrication of precise optical elements based on thin crystalline and glass substrates.

Preparation of ultrasmooth surfaces is one important issue for the fabrication of refractive x-ray optical components for synchrotron radiation instrumentation. In order to fabricate ultrasmooth surfaces in-house, some R&D projects have been carried out:

- A semiclean polishing laboratory, with three types of polishers, including a polisher with chemical-mechanical polishing (CMP) capabilities is now totally functional. We have purchased a Zeiss microscope for visual characterization of surfaces between polishing stages.
- A review of recent publications on fabrication of ultrasmooth surfaces has been shown that CMP is currently the best technique for this purpose. The most common polishing compounds being used for CMP processes are different types of colloidal liquids, but still there are not enough research results to fully understand the polishing mechanisms in many cases.

In order to obtain some ideas of how colloidal particles work in the polishing processes, we have chosen three type of commercially available slurries and analyzed them to get information about their chemical contents. Important from the point of view of polishing results is that none of these compounds contains chemicals that can etch silicon or germanium in free forms. A review of recent publications on the chemistry and technology of colloidal polishing compounds in combination with our experimental results allowed us to better understand the process and mechanism of CMP. By solving many other technical and technological problems and by applying a float polishing technique, we have been able to develop in-house the technology for ultasmooth polishing of silicon and have reached a 1 Å rms finish. However, we still have a lot to do towards fabrication of smooth and flat optical elements, and we are continuously working on development of better technologies for fabrication of x-ray optical elements.

Recently, fabrication of a Si crystal designed for sagittal focusing in a bender with a thickness of 0.9 mm over a diffraction area of 53 mm by 76 mm and with a thickness variation of only 5 microns over the entire diffraction area was achieved.

2.3.4 X-ray Characterization Laboratory

In accordance with its mission, the x-ray laboratory has served the APS users by offering them opportunities to test their crystals or thin layers deposited on different substrates. A single-axis diffractometer, a Laue camera, and a double-axis diffractometer (all installed at conventional x-ray generators—Rigaku and Spellman) were used to obtain the crystallographic orientation and/or to test numerous crystals. The instruments were available to all authorized users. However, most of the efforts in the x-ray lab were connected with diffractometers installed at the rotating anode generator, i.e., with the Topo Test Unit (TTU) and the triple-axis diffractometer.

In the past two years, a total of 122 crystal tests were performed with the use of the TTU. Of those, 90 tests were for silicon [predominantly monochromators and analyzers manufactured by the former User Program Division (UPD), and sometimes ingots to be used for fabrication], 4 for germanium, and 28 for diamond crystals. A majority of the tested optical elements (74) originated from XFD and UPD. However, 48 experiments were carried out for users from UNI-CAT, IMM-CAT, COM-CAT, SBC-CAT, Bio-CAT, Rockefeller University, Northwestern University, University of Illinois at Urbana Champaign, and the National Institute of Standards and Technology.

As in previous years, the triple-axis diffractometer was mostly used for reflectivity measurements (11 single- or multilayers were investigated) and for testing samples that were later used at the beamlines (about 12 different samples were checked).

Parallel to serving users, a major effort to rebuild and modernize the TTU was undertaken, including having easily exchangeable CCD and scintillation detectors, improving sample crystal rotations, and improving changing of monochromators.

2.3.5 Refractory Lenses

Focusing or collimating diverging x-ray beams produced by synchrotron sources is necessary in a range of applications. This can be achieved by using mirrors, crystals, zone plates, capillaries, and more recently by using a compound x-ray lens (CXL), proposed a few decades ago and more vigorously pursued since the mid-1990s.

With the reduction to practice of CXLs in the mid-1990s and the general realization of their usefulness, a number of attempts have been made to manufacture such lens arrays in a variety of suitable materials and configurations.

Recently, the APS has developed parabolic lenses in aluminum using microextrusion technology. With this technique, we are able, for the first time, to generate miles of x-ray lenses at a very economical cost and with reasonable precision. The first generation of such lenses has been designed, fabricated, assembled, and tested at the APS. Our first prototype lens of this kind is shown in Fig. 2.27.

The variable focus aluminum lens has been used at the APS to collimate a monochromatic, 81-keV undulator beam. Results indicate collimation consistent with theoretical expectations. At this energy, the absorption coefficient for aluminum is $\mu = 0.53976\ cm^{-1}$.

Two sets of lenses were tested on the APS 1-ID beamline. In one experiment, an upstream vertical slit was used to provide an approximately 0.6-mm-high beam at 35 m from the source where the CXL would be located. After inserting the lens, the transmitted beam through the lens was measured both 0.3 m and 24 m downstream using two vertical scans. Figure 2.28 shows the results.

The narrowing of the x-ray beam seen in both curves in Fig. 2.28 is due to two different effects. The curve for the beam at 0.3 m from the lens indicated not focusing—but the higher absorption of x-rays away from the optical axis of the lens, where the beam has to go through thicker aluminum layers. Focusing is apparent from the curve showing the measurements at 24 m from the lens. The beam is considered collimated where the two curves cross, in this case at about 160 lenses, in agreement with theory.

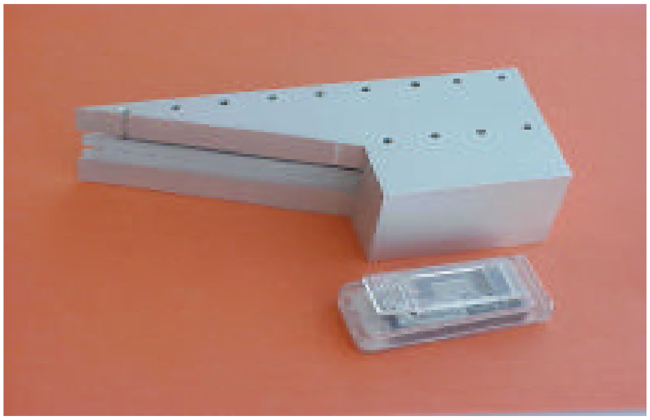

Fig. 2.27. A prototype variable focus compound x-ray lens. By moving the assembly horizontally, the incident beam from the right-hand side is made to pass through different numbers of lenses.

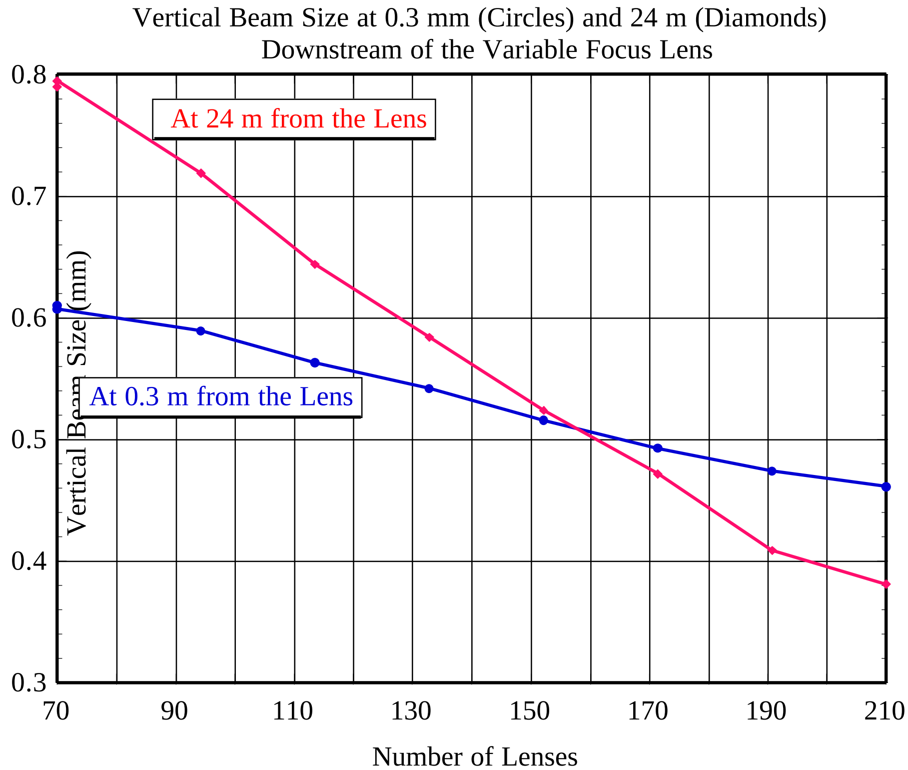

Fig. 2.28. Variation in the vertical size of an x-ray beam with the number of lenses on its path measured at 0.3 m and 24 m downstream of the CXL.

We expect that reducing the lens wall thickness from the present 0.2 mm to 0.1 mm will approximately double the throughput in the present setup. The next generation of the lenses, with a wall thickness of 0.1 mm has recently be obtained, and tests are planned.

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